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Synthesis, Properties of Derivative N, N[1] – Hexamethylene Bis - [(Methanol) -Carbamate] and Its Application.

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ABSTRACT: The proposed article relates to organic chemical synthesis and the study of derivatives of N, N[1]-hexamethylene-bis [(methanoyl) -carbamates]. Chemical properties and reactions of dichlorination, dinitrozozation, dibenzylation were studied. The results of the biological activity of derivatives of N, N[1]-hexamethylene-bis [(methanoyl) carbamates] were obtained. A stimulating effect on seed germination depending on concentration was established. The Ways of practical application of derivatives N, N[1]-hexamethylene-bis [(methanoyl) -carbamate] are outlined.

KEY WORDS: Organic synthesis, Derivatives, Carbamate, Hexamethylene, N,N[1]- dichlorination, dinitrozozation, dibenzylation, Field test.

I.INTRODUCTION

In chemistry and technology of synthetic organic compounds, the directions of fine organic synthesis of substanceshas acquired particular development, among which a significant role is given to the derivatives of carbamate and bis-carbamate derived from isocyanates, as well as hydroxyl containing radicals.

II. SIGNIFICANCE OF THE SYSTEM

The paper mainly focuses on how the chemistry derivatives of hexamethylene-bis [(methanoyl) -carbamates compounds. The study of literature survey is presented in section III, Proposed methodology and discussion is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and Conclusion.

III. LITERATURE SURVEY

Numerous studies in the field of derivatives of carbamates and bis-carbamates, currently underway, are stimulated not only by theoretical but also by practical needs. From this point of view, derivatives of carbamates and bis-carbamates are of undoubted interest as substances with different technical, biological and pharmacological activity. They are successfully used in almost all sectors of the national economy, in particular, in engineering as rubber vulcanization accelerators, as thermal stabilizers of polymers, additives to lubricating oils, and are used as starting products for the production of polymers, as corrosion inhibitors [1-9].
In agriculture they have been used as herbicides, fungicides, pesticides, defoliants, insecticides, nematocides, bactericides, biostimulants, and many others. The use of these class of compounds in medicineis of particular interest, as antitumor, antiviral, antidiabetic, reducing bad cholesterol, antiarrhythmic, anti-inflammatory and other drugs [10-21].

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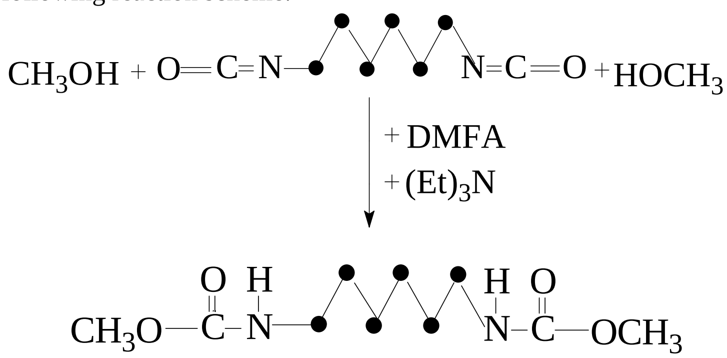
IV. PROPOSED METHODOLOGY AND DISCUSSION

The object of the study was the derivatives of N, N[1]-hexamethylene-bis [(methanoyl) -carbamates]. The course of the reaction and the individuality of the compounds are monitored by TLC on aluminum oxide of (II) degree of activity with the appearance of spots by iodine vapor. IR spectra recorded on a spectrometer VR-20. Laboratory tests have been conducted to identify the growth-promoting activity of N, N[1]-hexamethylene-bis - [(methanoyl) -carbamate] compounds. Their physical and chemical properties were studied.
In this regard, we presented previously conducted research in the field of the synthesis of new derivatives of N, N[1]-hexamethylene-bis [(methanoyl) -carbamates] and carried out a study of their chemical properties in the reaction centers.
So, by reacting methanol with diisocyanates, N, N[1]-hexamethylene-bis [(methanoyl) -carbamate] derivatives were obtained according to the following reaction scheme:



I

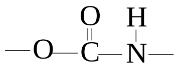
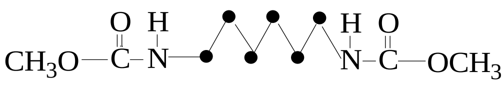
The reaction of hexamethylene diisocyanate with methanol was carried out at a molar ratio of reagents of 1: 2 at room temperature of 27-36 ° C for 3 -3,5 hours. As a result of the reaction, N, N[1]-hexamethylene-bis [(methanoyl) -carbamate] (I) is formed, which is a snow-white high-melting powder, difficultly soluble in water and other non-polar light solvents, which confirms the presence of two () -carbamate as well as hexamethylene hydrocarbons.

Table 1.Physico-chemical characteristics of N, N[1]-hexamethylene bis [(methanoyl) carbamate] (I)

Structural formula	Yield, %	MT, °C	R_f	Brutto formula	Elemental analysis, %						M_M
					Calculated			Found			
					C	H	N	C	H	N	
	99,2	1-3-104	0,74	$C_{10}H_{20}N_2O_4$	51,72	8,62	12,06	51,61	8,47	11,88	232

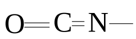
As can be seen from table (I), the yield of N, N[1]-hexamethylene-bis [(methanoyl) -carbamate] is rather high.The high yield of the resulting bis [(methanoyl) carbamate derivative] is apparently due to the high density and easy mobility of the electron cloud of the conjugated () group, which leads to an increase in the positive charge on the carbon atom of the isocyanate group, having an attack on this nucleophilic agent, as well as steric hindrances.
The structure of the synthesized compound (I) was established by the methods of IR and PMR spectroscopy and elemental analysis data (table 2).

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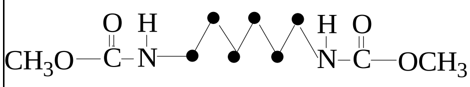
Table 2. IR and PMR spectral data of compounds (I)

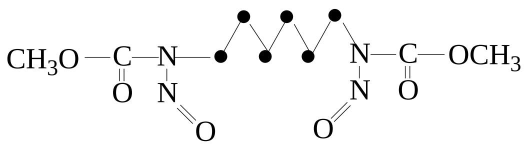
Compound I	IR-spectra, ν, sm^{-1}					PMR spectrum, δ, m.g.		
	–O–C(=O)–N(H)–	H–N–CH_2–	–C(=O)–	–N(H)–	$(CH_2)_6$	–CH_3	–N–CH_2–	–CH_2–
	1592	1430-1376	1690	3290	754-718	2,19	3,05	1,42-1,40

To identify the reactivity of N-H reaction centers of N, N^1-hexamethylene-bis [(methanoyl) -carbamate], we carried out the reactions of N, N^1-dinitrozozation, metallation, alkylation and halogenation.

A. Preparation of N, N^1 – dinitroso substituted -N, N^1-hexamethylene bis [(methanoyl) carbamate].

As a result of the reaction of N, N^1-dinitrosis of the bis [(methanoyl) -carbamate derivative] sodium nitrite (in excess) in formic acid, the corresponding N, N^1-dinitroso-substituted bis [(methanoyl) -carbamate] was obtained with a yield of 83,3 % (table 3).

Table 3. Physico-chemical parameters of the compound (II).

Structural formula	Yield, %	MT, °C	Brutto formula	Elemental analysis, %						M_M
				Calculated			Found			
				C	H	N	C	H	N	
	83,3	300 (decom)	$C_{10}H_{20}N_2O_4$	41,38	6,20	19,31	41,25	6,07	19,13	290

N, N^1- dinitrozozation proceeds by the mechanism of electrophilic substitution (S_E).

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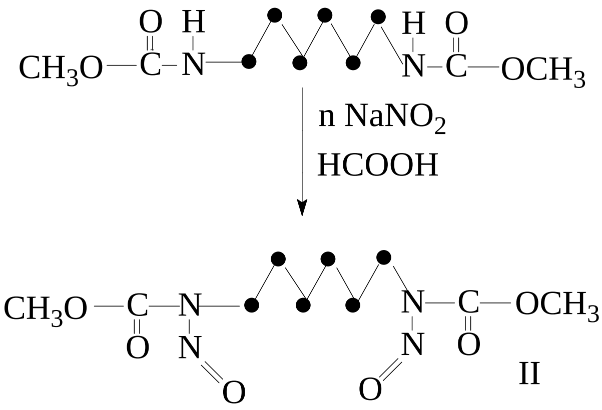


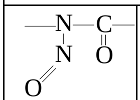
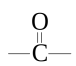
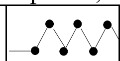
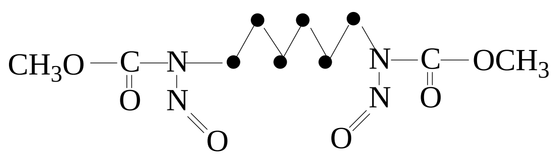
II

The attacking agent is nitrosonium ion $\overset{\oplus}{NO}$. Since nitrous acid, which is the most common agent, does not exist in its free form, sodium nitrite and a strong acid are used to carry out the process, attaching a proton, generates an ion $\overset{\oplus}{NO}$.

$$NaNO_2 + HCOOH \longleftrightarrow H_2\overset{\oplus}{NO_2} \longleftrightarrow \overset{\oplus}{NO} + HCOONa + H_2O$$

N, N[1]-dinitrosis is carried out with cooling (0-5 ° C) of the reaction mixture. Increasing the temperature is undesirable because it reduces the yield of the target product, and sometimes affects the direction of the reaction.
The identification of N, N[1] -dinitroso compounds is carried out in the N-nitrosogroup absorption bands. A very strong absorption band in the region of 1528-1440 cm^{-1} for groups is very characterized.
The structure of the synthesized compound (II) was established by IR spectroscopy and elemental analysis data (table 4).

Table 4.IR spectral data of compound (II).

Structural formula	IR-spectra, ν, sm^{-1}				
				$-CH_2-$	$-CH_3$
	1560-1430	1716	768-724	2870	2995

B. Preparation of N, N[1] disodium of substituted N, N[1]-hexamethylene bis [(methanoyl) carbamate].

One of the methods of metalation, which can be carried out using N – metalation, is the replacement of hydrogen atoms by sodium in the N–H group. The N, N[1]-hexamethylene bis [(methanoyl) carbamate] issubjected to targeted metallization of N-H groupswith CH_3ONa. The reaction of N, N[1]-dimetallization proceeds as follows:

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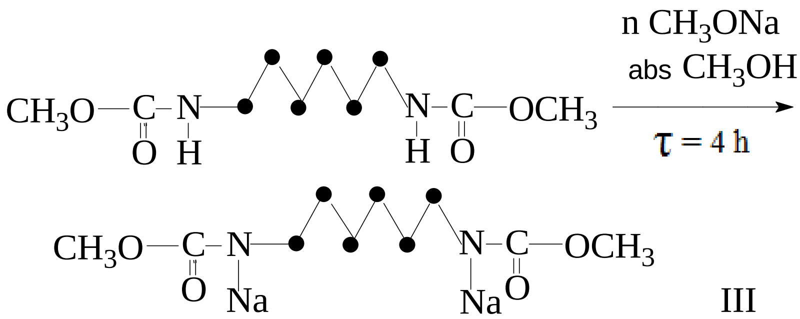
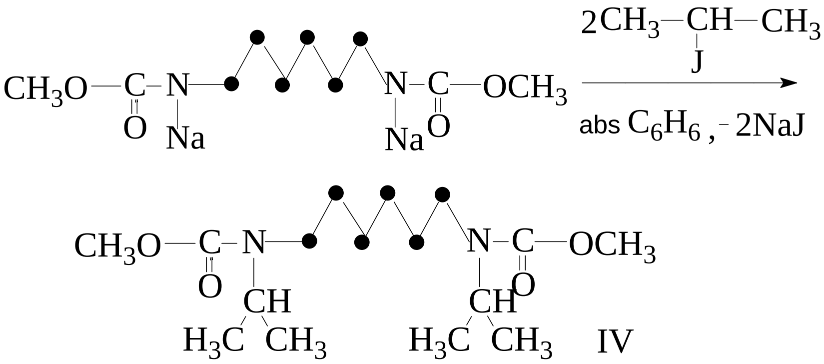


III

C. Preparation of N, N[1]-diisopropyl substituted N, N[1]-hexamethylene bis [(methanoyl) carbamate] (IV).

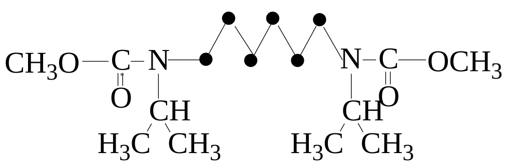
Isopropylation in carbamates with alkyl halides is of undoubted interest in determining the reactivity of N – H containing compounds. We carried out alkylation reactions, by the interaction of N, N[1]-disodium of derivatives of N, N[1]-hexamethylene-bis [(methanoyl) -carbamate] with iodide isopropyl in absolutely dry benzol at room temperature 28-31 ° C with stirring for 3,0-3,5 hours according to the scheme:



IV

The course of the alkylation reaction exclusively on the nitrogen atom N, N[1] - is explained, apparently, by the relatively easy dissociation of sodium in this atom due to the presence of carbonic groups in the neighboring. The yield of the product (IV) is 88,6 %, Mp = 157-158 °C.
Physico-chemical parameters of the obtained product (IV) are given in table 5.

Table 5.Physico-chemical parameters of the compound (IV)

Structural formula	Yield, %	MT, °C	R_f	Brutto formula	Elemental analysis, %		M_M
					Calculated	Found	
					N	N	
	88,6	157-158	0,72	$C_{16}H_{32}N_2O_4$	8,86	8,73	316

D. Preparation of N, N[1]-dichloro-substituted N, N[1]-hexamethylene bis [(methanoyl) carbamate] (V).

An efficient, affordable, cheap, environmentally friendly method for the implementation of N, N[1]-dichlorination of a bis-carbamate derivative by calcium hypochlorite on wet Al_2O_3 has been developed. The chemical reaction scheme is as follows:

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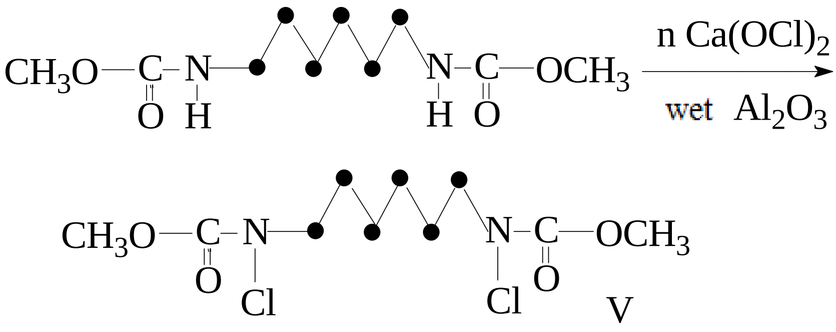
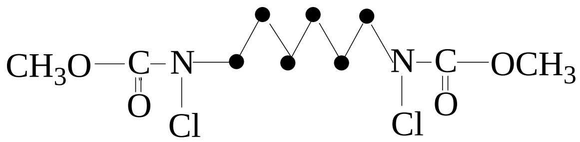


V

These reactions are of interest to many specialists chemists, pharmacologists, biologists, biochemists, bioorganics, microbiologists, and many others, because of the presence of a vital, highly reactive center (-N-H group) in the bis-carbamate derivative necessary for carrying out nucleophilic and electrophilic substitution.
The yield of the product (V) and physico-chemical parameters are shown in table 6.
To prove the structure of the newly synthesized N, N^1-dichloro- substituted -N, N^1-hexamethylene-bis [(methanolo) -carbamate], IR spectra were taken, elemental analysis and qualitative reactions with $AgNO_3$ were carried out.
Thus, methods have been developed for the preparation of N, N^1-hexamethylene bis [(methanoyl) carbamate] and its chemical properties have been studied for the N–H reaction centers: the reactions of nitrozozation, metallation, alkylation and chlorination.

Table 6.Physico-chemical parameters of the compound (V).

Structural formula	Yield, %	MT, °C	R_f	Brutto formula	Elemental analysis, %				M_M
					Calculated		Found		
					N	Cl	N	Cl	
	93,7	103-104	0,69	$C_{10}H_{18}Cl_2N_2O_4$	9,30	23,59	9,24	23,42	301

V. EXPERIMENTAL RESULTS

A. Synthesis of N, N^1-hexamethylene bis [(methanoyl) carbamate] (I).

7 ml of triethylamineare added to 16.0 ml (0.5 mol) of methanol, 40 ml. DMFA, while stirring, 42,4 ml (0,25 mol) of hexamethylene diisocyanate dissolved in 40 ml of DMFA are added dropwise at room temperature. The reaction mixture is stirred for 3 hours at a temperature of 39-42 °C. After the time the contents of the flask is transferred into a glass, water is added. The precipitate is washed with TLC. After drying, a colorless powder is obtained, the yield of the product (I) is 57,9 g (99,2 % of the theoretical); Mp = 103-104 ^{o}C.
Found, %: C 51,61; H 8,47; N 1,88
Calculated for $C_{10}H_{20}N_2O_4$, %: C 51,72; H 8,62; N 12,06

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B. Synthesis of N, N^1-hexamethylen-N, N^1-dinitroso-bis [(methanoyl) -carbamate] (II).

While constantly stirring, in excess for 3,5-4 hours, at a temperature of 0-5 ^oC, 0,6 g of sodium nitrite is added in portions to 2,32 g (0,01 mol) (II) dissolved in 75 ml of formic acid. After completion, the mixture is poured into a glass, water is added, the precipitated precipitate is filtered off, washed with benzol and dried, TLC is on Silifol plates, the yield is 83,3 %; Mp = 300 °C (decomp).
Found, %: C 41,27; H 6,07; N 19,13
Calculated for $C_{10}H_{18}N_4O_6$, %: C 41,38; H 6,20; N 19,31

C. Synthesis of N, N^1-disodium-N, N^1-hexamethylene bis [(methanoyl) carbamate] (III).

2,32 g (0,01 mol) of (I) is added in CH_3ONa (out of 0,031 g / mol and 80 ml of absol. CH_3OH). The mixture is stirred for 2 hours at a temperature of 20 °C and 2 hours at 40 °C. The precipitate is filtered off, washed with absol. CH_3OH and (III) is obtained, yield – 2,45 g – 89 % (from theoretical); Mp = 310 °C (decomp).

D. Synthesis of N, N^1-diisopropyl-N, N^1-hexamethylene-bis [(methanoyl) -carbamate] (IV).

2,45 g (0,08 mol) (III) are placed in 12 ml of DMFA, 3,5 ml (0,02 mol) of isopropyl iodide are added there dropwise with stirring, the mixture is stirred for 10 hours while heating in a boiling water bath, cooled and washed with 20 ml of water, the precipitate is separated, recrystallized from 50% alcohol, dried and (IV) is obtained with a yield of 2,72 g – 86,6% (from theoretical); Mp = 157-158 ^oC (decomp).
Found, %: C 60,57; H 10,04; N 8,73
Calculated for $C_{16}H_{32}N_2O_4$, %: C 60,75; H 10,12; N 8,86

E. Synthesis of N, N^1-hexamethylen-N, N^1-dichloro-bis [(methanoyl) -carbamate] (V).

2,32 g (0,01 mol) (I) are placed in 50 ml of CCl_4, 18 g of wet alumina and 4,0 g of calcium hypochlorite are added dropwise at a temperature of 40 °C for 1 hour. The reaction mass is left for 21 hours. Then, it is filtered, washed with ether, alcohol, dried and (V) is received with the release of –2,88 g (93,7 % of the theoretical); Mp = 103-104 ^oC.
Found, %: C 39,77; H 5,82; N 9,24; Cl 23,42
Calculated for $C_{10}H_{18}Cl_2N_2O_4$, %: C 39,86; H 5,98; N 9,30; Cl 23,59

Tests on the growth-promoting activity

To identify the growth-promoting activity of the compounds N, N^1-hexamethylene-bis - [(methanoyl) -carbamate] with the conditional name (AGM-XM-1), the tests were carried out in the laboratory of the Institute of Plant Chemistry of the Academy of Sciences of Uzbekistan,vegetable and cotton seeds served as biotests(17-21).
The experiments used cucumbers of the variety “Uzbekistan-740”, tomatoes of the variety “Temp” and medium-fiber cotton of the variety “S-6524”. The preparations were dissolved in DMF and used by the method of presowing seed locking for 18-20 hours. Were used concentration – 0,1; 0,01; 0,001; 0,0001 and 0,00001 %. The repetition of experiments was 4-fold. Accounting was carried out by measuring the length of the stem and root in 10 day old seedlings of cotton.
It was noted that all drugs tend to stimulate the growth of the root system of young seedlings, both vegetable crops and cotton.
Primary screening was carried out according to the method of Yu.V. Rakitin. This method allows you to quickly determine the degree of physiological activity of new chemical compounds, which is detected by stimulation or germination of plant seeds, as well as by changing the length of the roots and the length of the stem part.
The preparations were tested by the method of locking seeds in solutions of different concentrations with following germination in Petri dishes. Control seeds were soaked in distilled water.
Each series of experiments is accompanied by control. In the control variants, only pure solvent is added to the nutrient medium.
The result of the experiments is recorded after 3,5,7 and 10 days after inoculation (Tables 7-9).

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Comparative tests also show that the test drug AGM-XM-1, that isa derivative of N, N[1]-hexamethylene-bis-[(methanoyl) -carbamate] from 7,5 to 75000 times less than the low concentration of our drug, showed a higher growth-promoting activity than the ROSTLIN drug used in many branches of agriculture in Uzbekistan.

Table 7. The effect of the drug AGM-HM-1 on seed germination and growth seedlings of cotton varieties "C-6524"

Experiences A drug	Concentration, %	Germination, %	Cotton	
			Root growth,%	Stem growth, %
Control - water	without	80,0	100,0	100,0
N, N[1]-hexamethylene-bis-[(methanoyl) -carbamate]	0,1	85,7	112,7	106,6
	0,01	83,8	111,6	110,3
	0,001	87,6	137,4	119,5
	0,0001	85,3	116,3	109,6
	0,00001	82,4	122,6	117,2
«Rostlin» (famous)	0,75-1,0	80,0	102,6	102,4

The preparation AGM-XM-1 on a cotton crop showed biological activity at a concentration of 0,00001 % (at a dilution of 75000 times). The root growth was stimulated by 122,6 %, and the stem growth was 117,3 % higher than the control and the well-known drug ROSTLIN (concentration 0,75-1,0 %).

The preparation AGM-XM-1 on tomatoes, similarly to previous cultures, showed a very high biological activity, 147,6 % at a concentration of 0,001 (even at a dilution of 750 times). The AGM-XM-1 preparation on a cucumber culture also showed biological activity at a concentration of 0,0001 % (i.e., at a dilution of 7500 times). The preparation contributed to the growth of the root of 137,5 %, slightly lower - the growth of the stem by 114,5 % above the control and the well-known drug " ROSTLIN " (concentration 0,75-1,0 %).

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Table 8. The effect of the drug AGM-HM-1 on seed germination and seedling growth Tomato varieties "Temp"

Experiences A drug	Concentration, %	Germination, %	Tomato	
			Root growth,%	Stem growth, %
Control - water	without	50,0	100,0	100,0
N, N[1]-hexamethylene-bis-[(methanoyl) -carbamate]	0,1	51,0	105,0	118,4
	0,01	59,7	119,3	125,7
	0,001	57,5	147,6	131,6
	0,0001	48,8	117,6	109,5
	0,00001	52,7	121,3	107,6
«Rostlin» (famous)	0,75-1,0	52,1	101,7	100,7

Table 9.The effect of the drug AGM-HM-1 on seed germination and growth of seedling s of cucumber varieties "Uzbekistan-740"

Experiences A drug	Concentration, %	Germination, %	Cucumber	
			Root growth,%	Stem growth, %
Control - water	without	100,0	100,0	100,0
N, N[1]-hexamethylene-bis-[(methanoyl) -carbamate]	0,1	100,0	109,3	108,6
	0,01	100,0	111,6	112,3
	0,001	100,0	122,7	111,6
	0,0001	100,0	137,5	114,3
	0,00001	100,0	131,3	119,7
«Rostlin» (famous)	0,75-1,0	100,0	103,4	101,7

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Thus, the low toxic (LD ≈ 4717 mg / kg) AGM-XM-1 preparation showed high stimulating properties on the seeds of tomato, cucumbers and cotton at 0,0001 and 0,00001 % of concentration. After the initial tests for the growth-promoting activity of the drug I (AGM-XM-1), field tests were recommended on S. Agzamov's farm in the Kasbinsk fog of the Kashkadarya region of Uzbekistan from April to November 2017-2018.

VI. CONCLUSION AND FUTURE WORK

Field tests for the growth-promoting activity of the drug AGM-XM-1

The resulting growth stimulator (I), in particular N, N^1 – hexamethylene bis- [(methanoyl) carbamate], was tested at a concentration of 0,0001 % (i.e., a dilution of 7500 times). «Temp» tomatoes, «Uzbekistan-740» cucumbers, «S-6524» medium-fiber cotton, corn and sunflower were used in the farm, on an area of 500 hectares. An additional 1338 tons of cotton were obtained, which is the estimated economic effect of about 600 million soums only for cotton growing. Similarly, good results were obtained on tomatoes, cucumbers, sunflowers and corn.
Thus, the preparation (I) (AGM-XM-1), recommended by us in a concentration of 0,001- 0,0001 %, surpasses many well-known drugs in biostimulating activity.

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Abdukhamid Gofurovich Makhsumov (born in 1936) created a scientific school in the direction of "Synthesis, properties and use of biologically active compounds." He published more than 1,800 scientific works, including 310 patents, more than 10 textbooks and teaching aids. He introduced more than 10 drugs in medicine, 115 biostimulators, herbicides in agriculture, animal husbandry, over 50 dyes in the textile, chemical industries, national economy.